

# MICROSTRUCTURE AND MECHANICAL PROPERTIES OF AL-AL<sub>4</sub>C<sub>3</sub> MATERIALS

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## Abstract

Dispersion strengthened aluminium compacts have been prepared by powder metallurgy. The base microstructure is aluminium matrix strengthened with dispersed ceramic particles. The strengthening is direct by dislocation movement retardation, and indirect by deformation induced microstructure modification in the next technological steps. The method of mechanical alloying process is described. Carbon transformation to carbide Al<sub>4</sub>C<sub>3</sub> is characterised for different heat treatment schedules and nine commercial carbon powders tested. The micromechanism of carbon incorporation into the metallic powder, and the compacting of it are described. The influence of dispersed carbides on mechanical properties is evaluated together with the influence of deformation on microstructure and properties. Ductility anomalies up to a type of superplasticity were observed at certain tensile testing strain rates.

## Introduction

A mode of mechanical alloying is reaction milling, developed for dispersion strengthened aluminium production [1]. To produce aluminium dispersoid the aluminium powder is intensively dry milled with carbon powder. The transformed dispersed phase Al<sub>4</sub>C<sub>3</sub> is then produced by a chemical reaction, which starts during milling, and it is completed at the next heat treatment process. The resulting powder mixture is then pressed, compacted and isostatic pressing and hot extrusion prepare the final compacts.

The aim of this paper is to study the influence of the various graphite types when mixed with Al powder, and heat treatment procedure on the microstructure and properties of dispersion strengthened aluminium type Al-Al<sub>4</sub>C<sub>3</sub>. The influence of carbides characteristics on mechanical properties is evaluated together with the influence of applied deformation mode on the microstructure development and mechanical properties.

## Experimental material and methods

The experimental material - dispersion strengthened aluminium with Al<sub>4</sub>C<sub>3</sub> particles, was prepared by intense milling of aluminium powder with different types of carbon, as shown in Tab.1. The prime aluminium powder grain size was 100 µm with the carbon content of 0.6 - 3 wt. %. The final carbide content was in the range of 2.5 - 12 vol.%. The obtained mixture was compacted at 600 MPa and thermally treated at 450, 500, 550, and 600°C whereas treatment times of 1, 3, 10, and 30 hours were employed. The final compacting by hot extrusion at a temperature of 550°C and a reduction rate of 94% on the cross section was applied [5]. The experimental material has been both prepared, and tested by gas chromatography for carbides Al<sub>4</sub>C<sub>3</sub> content, at the Institute for Chemical Technology of Anorganic Materials, TU Vienna.

Tab.1 Types of different carbon types used

Notation	Type	Commercial Carbon	Notation	Type	Commercial Carbon
A	a <sub>1</sub>	LTD	F	a <sub>2</sub>	Farbruss FW 2
B	a <sub>1</sub>	Spezialschwarz 5	G	a <sub>2</sub>	Flammruss 101
C	a <sub>1</sub>	Spezialschwarz 500	H	c	Thermax
D	a <sub>1</sub>	Printex 30	I	b	Grafit KS 2.5
E	a <sub>2</sub>	Printex 400			

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### Carbonization kinetics

For the employed nine types of commercial carbon system Al – 8Al<sub>4</sub>C<sub>3</sub> labelled A to I correlation were sought between physical and chemical properties and milling parameters, or carbide transformation rate, and properties of the produced compacts.

The different carbon types showed different distributions of carbon in the aluminium powder. Their susceptibility to milling was measured by the ability to prepare homogeneous distribution without being formed clusters. According to the obtained results the carbon forms were divided into 4 types:

a<sub>1</sub>) porous types of furnace black, made by uncomplete burning of carbohydrates at low temperatures, with very good properties. They are fine, with high contact surface, and easy destruction of clusters.

a<sub>2</sub>) porous types of furnace black, made by uncomplete burning of carbohydrates at higher temperatures. They are fine, but they form more stable clusters, resistant to desintegration.

b) electrographite, with layered structure, with good susceptibility to milling, though coarse grained and with smaller contact surface; comparable to furnace black (a<sub>1</sub> and a<sub>2</sub>)

c) cracked carbon, forms strong clusters, and the carbon to carbide transformation rate is low.

The milling kinetics of the system is described in more detail in reference [6]. From the results, the homogeneity of carbide distribution and contact surface area influence the Al+C transformation kinetics to Al<sub>4</sub>C<sub>3</sub>. The dependence of the transformation rate on temperature and hold time for the 4 carbon types is shown in Fig.1. The good susceptibility to transformation for porous furnace black (a<sub>1</sub> and a<sub>2</sub>) and that of electrographite (b) is evident.

The dependence of tensile strength and elongation on carbon content (wt.%) for Flammrus LTD is presented in Fig.2.

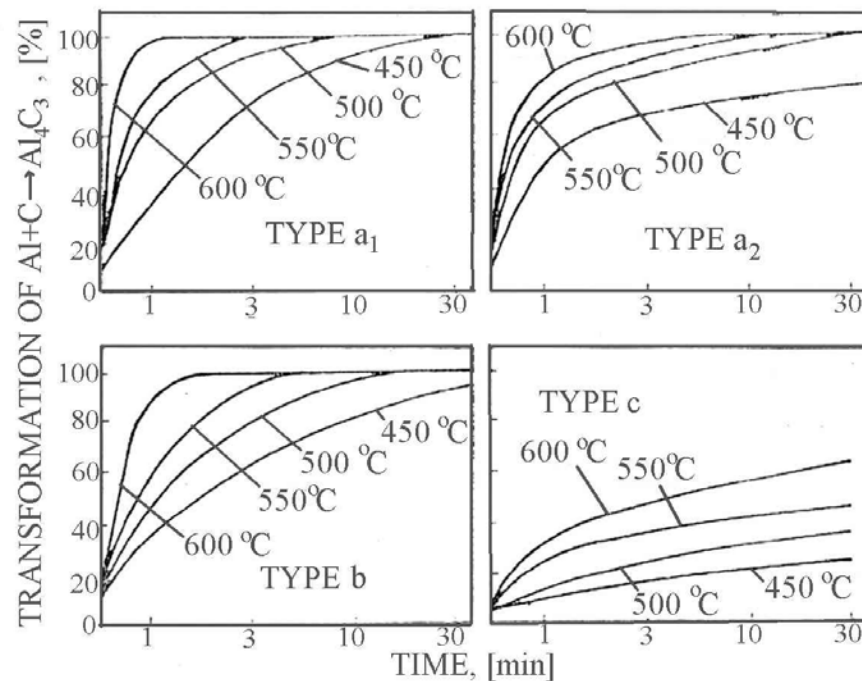


Fig.1 Dependence of carbon to the carbide transformation rate on heat treatment temperature and hold time for four carbon types.

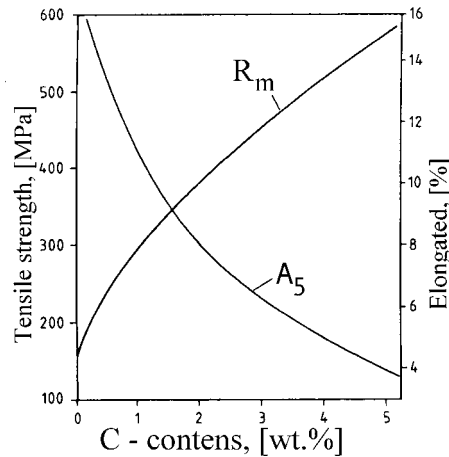


Fig.2 The dependence of tensile strength and elongation on carbon content (wt.%) for Flammrus LTD

### Microstructure and mechanical properties

Light microscopy microstructure analysis of the produced compacts proved a high homogeneity of dispersed particle distribution in the direction perpendicular to the direction of hot extrusion. In the longitudinal direction of the bar as a result of hot extrusion the  $Al_4C_3$  carbide particles were arranged into bands.

Electron microscopy analysis was conducted using carbon replicas and thin foils. The carbon replicas were not of help for quantitative evaluation. Transmission electron microscopy of thin foils offered better results. For all the tested carbon combinations from the A to I labels, thin foils were produced for the heat treatment  $450^\circ C/30$  h. The  $Al_4C_3$  particle size and the subgrain size were measured using the thin foils. The dispersed phase  $Al_4C_3$  particle size was measured on 200 to 300 thin foil structures, and it was constant and as small as 30 nm. The particle size was influenced neither by the carbon type nor by the heat treatment technology applied.

The mean distance between the particles depended strongly on the carbon type, as it depends on the efficiency of transformation. Subgrain size measured in the range of 100 grains in thin foils depended on the carbon type, as well. It ranged from 0.3 to  $0.7\ \mu m$ . The stability of properties, resulting from graphite type I (KS 2.5), led to the highest production and utilization of this type of dispersion strengthening. The results on mechanical behaviour of the compacted system, listed in the next parts of this paper are for this material selected.

In our previous works [8, 9, 10], we have evaluated the distance between the particles by point object simulation methods. The example of four interparticle distance categories is shown in Fig.3. This includes the mean interparticle distance  $\lambda_\mu$ , the mean minimum distance  $\lambda_\rho$ , the mean visibility  $\lambda_v$ , and the mean free spherical contact distance  $\lambda_g$ . The characteristics and properties of these parameters have been analyzed in [9].



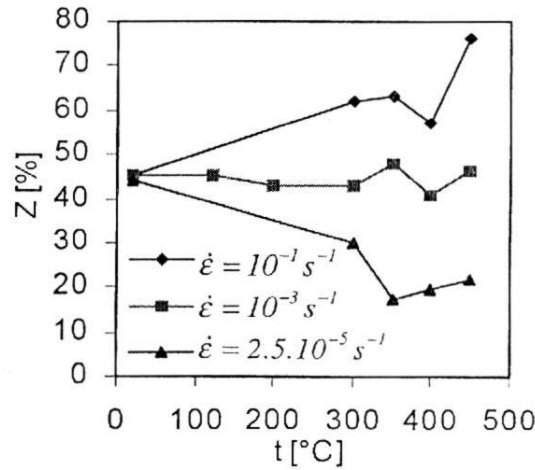


Fig.5 Reduction of area Z as a function of strain rate and temperature.

The Al-Al<sub>4</sub>C<sub>3</sub> system with 4 vol. % of Al<sub>4</sub>C<sub>3</sub> was tested under different tensile conditions, where three different strain rates and different testing temperatures up to 450°C were used [12]. The deformation mechanism and fracture mechanism were analyzed corresponding to different testing conditions. For the higher strain rates of 10<sup>-1</sup>s<sup>-1</sup> at 450°C, a significant growth of plastic properties was observed. The high uniform elongation A<sub>5</sub> of the specimen gauge length, and corresponding reduction values of the reduction in area Z were manifested in Fig.5. The ductility anomalies are showing an onset of a type of superplasticity. On the other hand, when testing temperature was of 450°C and low strain rates 10<sup>-5</sup>s<sup>-1</sup>, the microstructure was polygonized. Sliding along grain boundaries, accommodated by dislocation creep, would be the prevailing mechanism of deformation and plastic properties are extremely low. According to [13] it was proved that for materials Al - 12Al<sub>4</sub>C<sub>3</sub>, the main mechanism responsible for superplastic behaviour is the grains rotation process and not sliding. As an evidence of this process, one may use the result on grain size and measurement shape control at longitudinal and transverse direction carried out in this foils. The mechanisms of grain re-arrangement in superplasticity deformation process by sliding (a) and by rotation (b) are documented on Fig.6. The fracture mechanism was dominantly intergranular.

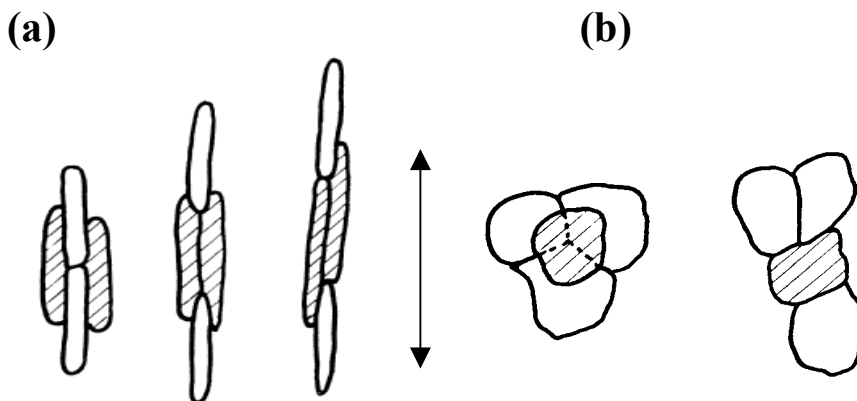


Fig.6 The mechanisms of grain re-arrangement in superplasticity deformation process by sliding (a) and by rotation (b)

Figure 7 shows the dependence of optimum superplastic strain rate on the inverse grain size for a large variety of superplastic aluminium alloys produced by different routes [14]. These dependencies suggest that the strain rates up to  $10^{-1} \text{ s}^{-1}$ , limited by the testing equipment, were out of the optimum conditions to get superplastic behavior.

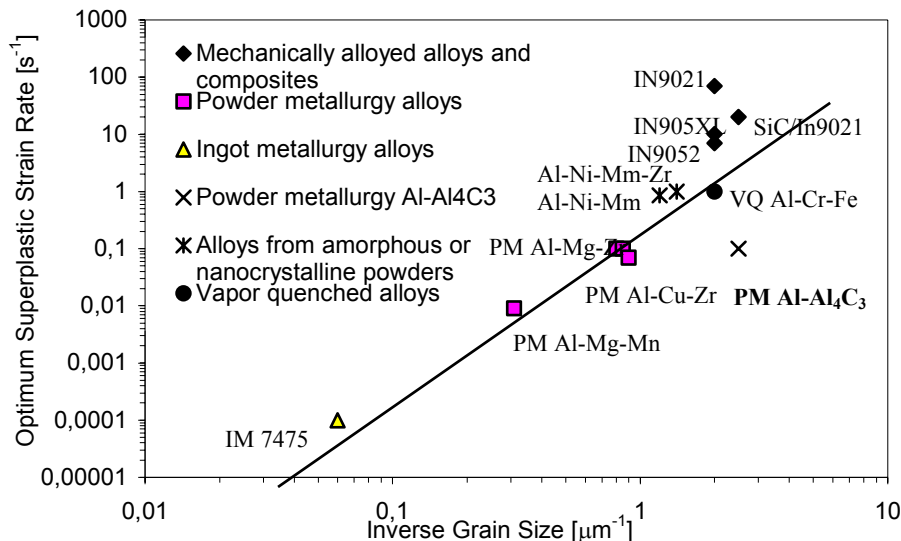


Fig.7 Dependence of optimum superplastic strain rate on the grain size for superplastic Al alloys produced by different routes.

## Conclusions

The obtained results on the mechanical alloying process and heat treatment of Al - C system, and on deformation behaviour of dispersion strengthened Al – Al<sub>4</sub>C<sub>3</sub> system prepared under different conditions, can be summarized as follows:

- It was shown that the transformation efficiency of carbon to Al<sub>4</sub>C<sub>3</sub> by heat treatment of aluminium with the porous furnace black a) and electrographite b) is higher, than that of the hard cracked graphite c).
- The volume fraction of carbide phase Al<sub>4</sub>C<sub>3</sub> and the efficiency of transformation are in good agreement with resulting microstructure and achieved mechanical properties.
- Microstructure and mechanical properties showed that the best strengthening is obtained with carbon types LTD (A) and KS 2,5 (I) with a high transformation rate, high Al<sub>4</sub>C<sub>3</sub> carbide content, and low subgrain size. On the other side, the strengthening resulted from the cracked Thermax (H) graphite is the lowest due to the low transformation rate  $\text{Al} + \text{C} \rightarrow \text{Al}_4\text{C}_3$
- The temperature dependencies of ductility, and reduction of area in temperature range of 350–450°C and strain rate of  $10^{-1} \text{ s}^{-1}$ , indicated a considerable increase of these properties. In a case when the volume fraction of Al<sub>4</sub>C<sub>3</sub> changes from lower to higher, the grain rotation mechanism dominates instead of the grain boundary sliding.

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